

PROJECT NUMBER: 6505  
PROJECT TITLE: Special Investigations/Methods Development  
PROJECT LEADER: D. F. Ingraham  
PERIOD COVERED: January, 1989

## I. PROJECT ART

A. Objective: Provide analytical support to project ART.

B. Results:

A Stabilwax-DB $\otimes$  column from Restek Corporation is currently being evaluated as an alternative to the J&W CAM column, due to long delays in receiving the latter columns. Thus far, the Restek column appears to be satisfactory for the GC determination of nicotine. The optimum linear carrier gas velocity for this analysis was determined to be about 30-32 cm/second, which should be applicable for any 15m x 0.25mm ID, 0.25  $\mu$ m film thickness column currently in use.

Monitor data collected at BHPP from runs numbered 258 to 279 were subjected to statistical analysis by Dick Jones. The high (unextracted) monitor showed an average value of 2.65% with upper and lower control limits of 2.76 and 2.54, respectively, and an estimated standard deviation of 0.05. The low (extracted) monitor showed an average of 0.068% with upper and lower control limits of 0.078 and 0.057, respectively, and an estimated standard deviation of 0.005. These data suggest that the extracted samples can be reported to three decimal places and the feed samples to two decimal places. The control charts showed a fairly random distribution around the mean for the low monitor, but several data points in a row above and below the mean for the high monitor.

C. Plans: Continue analytical support on an as needed basis. Continue to run and analyze the monitor data at BHPP. Investigate any analytical differences between the 5880A and the 5890A GC's.

## II. ANALYSIS OF RESIDUAL SOLVENTS IN PACKAGING MATERIAL

A. Objective: To provide headspace analyses for residual solvents from packaging materials and develop a QA method for the routine analysis of packaging materials.

B. Results:

Approximately 20 samples were analyzed by GC/HS this past month for residual solvent content. Several of these samples were also run by GC/FT-IR/MS for confirmation or identification of unknown solvents.

Phone support and a sample of our standard was provided to one of our vendors (Golden Belt). A standard solution was also provided to QA. Don Simmons (QA) was given a quick introduction to the

GC/HS analysis and provided with sufficient information to get started when his equipment arrives. Don will be assuming responsibility of analyzing the routine samples in QA.

- C. Plans: We will continue to analyze non-routine samples and provide support to QA as needed, including further training of the analyst in QA.

### III. MATERIALS EVALUATION

- A. Objective: To identify components of commercial products prior to their use at PM facilities.
- B. Results: Samples analyzed this month included cleaners, machine parts, lubricants, tube belts, and metal coatings. Recommendations were made for all samples.
- C. Plans: The analysis of samples for the audit program will begin after receipt of the January report from Purchasing.

### IV. RESPONSE TO ANALYTICAL REQUESTS

- A. Objective: To provide analytical support to R&D and Operations personnel and projects.

B. Results:

Analyses and investigations by project personnel during the month of January included:

Several customer complaint samples were analyzed for possible contaminants.

Approximately 100 filter plug samples were analyzed for water, triacetin, and menthol. These plugs, which were submitted by T. Van Auken, contained different levels of triacetin and had been exposed to menthol vapors at different relative humidities. The results showed that both water and triacetin content had an effect on the level of menthol observed, but a simple model could not explain all the data. These data were reported to the submitter for further analysis.

Anethole and ethanol content of Marlboro cigarettes were determined for the Marlboro Standardization Program.

A sample of paraben was received from Park 500 which was determined to have an off odor. Compared to an acceptable control, the sample was found to contain 2,2,4-trimethyl-1,3-dioxolane.

Several tobacco samples were analyzed for calcium by titration. The titration method allowed a faster turnaround and relieved the workload on the AA, where this analysis had been performed in the past.